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**THE EFFECT OF MANGANESE PHOSPHATE
COATINGS ON FATIGUE CRACK INITIATION**

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**RICHARD FARRARA
JOHN H. UNDERWOOD**

JUNE 1990



**US ARMY ARMAMENT RESEARCH,
DEVELOPMENT AND ENGINEERING CENTER
CLOSE COMBAT ARMAMENTS CENTER
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20. ABSTRACT (CONT'D)

Lowering the yield strength of the A723 material via raising the tempering temperature reduces the deleterious effect from the phosphating process because the material becomes less and less sensitive to stress concentration.

Shot peening the surface of the A723 material eliminates the effect of the phosphating process on the crack initiation life.

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INTRODUCTION

The objective of this investigation was to determine the effect of manganese phosphate coatings on the fatigue crack initiation life of A723 high strength steel.

A common method for protecting the surface of steel from corrosion is to coat the surface with manganese phosphate prior to applying a supplementary coating such as oil or solid film lubricant. The protective benefits from phosphate coating are well-documented (ref 1). However, since the phosphating process can result in a rough and pitted surface, the potential exists for premature cracking and degradation in the fatigue life of a phosphate-coated component. Since the major portion of the total fatigue life of many components, including cannon components, is the number of cycles required to initiate a crack, any cause for premature crack initiation is a major concern.

A comprehensive literature review did not provide any information on the effect of phosphate coatings on fatigue cracking. Therefore, there are a number of questions which remain unanswered.

1. To what degree does manganese phosphate degrade the crack initiation life of A723 high strength, quenched, and tempered steel?
2. Shot peening the surface creates a residual compressive stress thereby improving the crack initiation life. Would shot peening prior to phosphating eliminate any deleterious effect of phosphate on the crack initiation phase of fatigue?
3. Is a surface left unprotected to corrosion, i.e., a rusted surface, more detrimental to crack initiation life than a manganese-phosphated surface?
4. Is the deleterious effect of manganese phosphate dependent upon the yield strength of the base material?

APPROACH TO THE PROBLEM

The approach used was to fatigue cycle three-point bend specimens made from A723 steel in various heat treated conditions. The specimens contained a semi-circular notch that was placed opposite the load in order to apply a tensile stress cycle to the notch root (see Figure 1). Details of the surface conditions, material conditions, specimen design, and the testing procedure follow.

1. Surface Conditions:

- Not coated with the 0.25-in. radius notch produced by milling (63 RMS maximum surface roughness).
- Manganese phosphate-coated with normal free acid (5.6 to 6.4 pts) and high free acid (9.0 pts.). Phosphating procedure and definition of points are outlined in Appendix A.
- Shot peened and not coated. Shot peening procedure is outlined in Appendix A.
- Shot peened and manganese phosphate-coated.
- Rusted with as-machined specimen subjected to 6 hours in a 5 percent salt spray chamber (ASTM B-117) to generate the rusted surface condition.

2. Material Conditions:

The material was A723 steel from a forging with the chemical composition listed in Table I. The average yield strength of the original forging used for evaluating the previously listed surface conditions was 170 Ksi. To determine if the effect of manganese phosphate is a function of the material's yield strength, the A723 steel (originally austenitized at 1550°F, water quenched, and tempered at 1075°F) was retempered at 1125, 1200, and 1350°F in order to decrease the yield strength levels, as shown in Table II.

3. Specimen Design:

The specimen was a notched rectangular beam that was loaded as a three-point bend specimen shown in Figure 1. This design controls where the crack initiates, allows straightforward stress and fracture mechanics analysis, is easily manufactured and fatigue cycled, and is small enough to provide batch processing for the shot peening and manganese phosphate processes.

4. Testing Procedure:

The specimens were fatigue cycled by loading the simply supported specimen at mid-length thereby applying a bending, tensile stress at the semi-circular notch. The loading was applied at 1 Hz by a servo-controlled electro-hydraulic machine that maintained the load range constant. Maintaining the constant load range causes rapid propagation after a crack initiates. Therefore, the number of cycles to break the specimen, our definition for failure, is only slightly more than the number of cycles to initiate a crack across the full width of the notch root.

The loading ranges were selected to generate failure in the low cycle regime (10,000 to 100,000 cycles). Calculations for determining the load ranges (800-lb. minimum load to 8000-lb. maximum--named "high load," 550-lb. minimum load to 5500-lb. maximum--named "low load") are explained in Appendix B.

RESULTS

The test data for the various surface conditions applied to the 170 Ksi yield strength specimens are listed in Table III. The test data for the uncoated and manganese phosphate-coated surface condition applied to specimens at the lower yield strengths are listed in Table IV. The data were statistically analyzed by applying the "Student" t test (ref 2) (two-tailed) with a 99 percent confidence limit in order to determine if there is a statistically

significant difference between results for various conditions. This analysis is summarized in Table V. The crack initiation life versus nominal stress at the notch root for the 170 Ksi yield strength specimens with various surface conditions is plotted in Figure 2. The effect of the material yield strength on crack initiation of not coated and manganese phosphate-coated specimens is plotted in Figure 3.

The following is a discussion of the effect of manganese phosphate, rusting, and shot peening on the crack initiation life of high strength A723 steel (Table III, Table V, and Figure 2).

1. Effect of Manganese Phosphate:

Comparing the average life of no coating versus manganese phosphate coating (Table III) reveals a drastic decrease in life if manganese phosphate is applied. The difference becomes greater as the applied stress decreases, which is revealed in Figure 2. The statistical analysis (Table V) reveals that t_{actual} is significantly larger than $t_{0.995}$ (99 percent confidence limit), hence, statistically there is a significant difference between the two groups. Comparing manganese phosphate produced by a normal free acid level (5.6 to 6.4 pts) versus a high free acid level (9.0 pts) reveals that the average life of high free acid phosphate was slightly higher than the average life of the normal free acid phosphated-specimen (Table III), however, statistically there is no significant difference. The degradation in life of the phosphated specimens is a result of pits (act as stress concentrations) in the steel surface created by the phosphating process. These pits can be seen in Figure 4.

2. Effect of Rusting:

Comparing rusted specimens to unrusted/not coated specimens reveals that the average life (Table III) of the rusted specimens was slightly lower

than the unrusted specimens, however, statistically (Table V) there is no significant difference. Comparing the rusted specimens to the manganese-phosphated specimens (Table III) reveals that the average life of the manganese-phosphated specimens was significantly lower than the average life of the rusted specimens, and statistically (Table V) there is a significant difference.

3. Effect of Shot Peening:

Comparing shot peened/not coated to shot peened/manganese phosphate-coated results (Table III and Figure 2) reveals that the shot peening effect overshadowed the manganese phosphate effect, i.e., there is no significant difference in the average lives (Table V). Also, comparing shot peened/not coated versus not coated/not shop peened (Table III and Figure 2) reveals that shot peening drastically increased the life, as could be anticipated.

The following is a discussion of the results for the effect of the material yield strength on the fatigue life of not coated and manganese phosphate-coated materials (Table IV and Figure 3). Comparing the data listed in Table IV and shown in Figure 3 reveals that as yield strength decreased the life of the specimen not coated decreased (expected), whereas the life of the manganese phosphate-coated specimens increased as yield strength decreased. In other words, the degradation in fatigue life due to the manganese phosphate coating becomes less severe as the material's yield strength decreases. This result is explained by the understanding that the notch sensitivity of steel is decreased when the yield strength is decreased.

CONCLUSIONS

1. Coating high strength, quenched, and tempered A723 steel with manganese phosphate will significantly reduce the crack initiation life and hence

reduce the fatigue life of the component. This reduction in life is caused by the pits generated in the surface of the base material by the phosphating process which act as stress risers (Figure 4).

2. The deleterious effect on fatigue life from manganese phosphate increases as the applied stress decreases (Figure 2).

3. The decrease in fatigue life from manganese phosphate is more significant than from rust.

4. Shot peening will significantly increase crack initiation life and the improvement becomes larger as the applied stress becomes less. Applying manganese phosphate to a surface that was previously shot peened does not reduce the fatigue life. If fatigue life and corrosion resistance are both important, the surfaces should be shot peened prior to applying manganese phosphate.

5. Reducing the material yield strength will decrease the fatigue life of components not coated, whereas the trend is the opposite if the surface has been manganese phosphate-coated. This occurs because the material becomes less sensitive to stress concentrations (pits) as its yield strength decreases. There is a lower limit of yield strength where cracking will initiate as quickly on a surface not coated as on a manganese phosphate-coated surface.

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2. M. R. Spiegel, Theory and Problems of Statistics, Schaum Publishing Co., New York, 1961, pp. 189-190.
3. J. H. Underwood, "Fatigue Life Analysis and Tensile Overload Effects With High-Strength Steel Notched Specimens," in: High Pressure in Science and Technology, Part II. Fluids, Engineering, and Safety," Elsevier Science Publishing Co., Inc., New York, 1984, pp. 209-214.

TABLE I. CHEMICAL COMPOSITION OF A723 STEEL

	Specified	Actual
C	0.32/0.36	0.33
Mn	0.55/0.65	0.55
Si	0.25 max.	0.22
P	0.010 max.	0.007
S	0.008 max.	0.002
Ni	2.1/2.25	2.11
Cr	0.9/1.1	1.09
Mo	0.45/0.55	0.47
V	0.09/0.12	0.10
Al	0.01 max.	0.007
Ti	0.015 max.	0.002

**TABLE II. TEMPERING TEMPERATURE AND MECHANICAL PROPERTIES
(Average of Two Tests)**

	Tempering Temperature (°F)	Yield Strength 0.1% Offset (Ksi)	Ultimate Strength (Ksi)	Reduction in Area (%)
Tube Forging	1075	170	184	48
Retempered	1125	160	170	50
	1200	130	142	60
	1350	122	199	17

TABLE III. EFFECT OF SURFACE CONDITIONS ON CRACK INITIATION FOR 170 KSI YIELD STRENGTH A723 STEEL

Surface Condition	Maximum Nominal Stress (Ksi)	No. of Specimens	Average Life (Cycles)	Standard Deviation (Cycles)
Not coated - not shot peened	117	10	50,530	12,430
Manganese phosphate-coated - not shot peened (normal free acid - 5.6 to 6.4 pts)	171	3	9,630	1,010
	117	15	19,340	2,560
	171	3	4,700	290
Manganese phosphate-coated - not shot peened (high free acid - 9.0 pts)	171	3	6,330	480
Rusted (6 hrs - 5% salt spray)	117	3	39,030	8,910
Shot peened - not coated	117	2	148,000	8,000
	171	2	15,150	1,350
Shot peened - manganese phosphate-coated	117	2	126,500	8,500
	171	2	12,900	900

TABLE IV. EFFECT OF YIELD STRENGTH ON CRACK INITIATION FOR RETEMPERED A723 STEEL - NOT COATED VERSUS MANGANESE PHOSPHATE-COATED

Yield Strength (Ksi)	Average Life	
	Not coated	Manganese Phosphate-Coated
160	66,970	24,700
130	42,330	28,600
122	30,130	30,130

- (1) Maximum nominal stress - 117 Ksi.
- (2) Three specimens per test condition.

TABLE V. "STUDENT'S" T DISTRIBUTION TEST OF FATIGUE DATA - VARIOUS SURFACE CONDITIONS OF 170 KSI YIELD STRENGTH A723 STEEL

	Load Level*	Degrees Of Freedom (N ₁ +N ₂ -2)	t _{actual}	t _{0.995}	Significant Difference
Not coated versus manganese phosphate-coated	Low	23	9.038	2.81	Yes
Manganese phosphate-coated normal versus high free acid	High	7	5.42	3.50	Yes
Not coated - rusted versus not rusted	High	4	4.115	4.60	No
Manganese phosphate-coated versus rusted	Low	11	1.37	3.11	No
Shot peened - not coated versus manganese phosphate-coated	Low	16	6.79	2.92	Yes
	Low	2	0.04	9.92	No
	High	2	0.28	9.92	No

$$t_{\text{actual}} = \frac{\bar{X}_1 - \bar{X}_2}{\sigma \sqrt{\frac{1}{N_1} + \frac{1}{N_2}}} \quad \text{where } \sigma = \sqrt{\frac{N_1 S_1^2 + N_2 S_2^2}{N_1 + N_2 - 2}}$$

\bar{X}_1 and \bar{X}_2 = mean life to failure of group 1 and group 2
 S_1 and S_2 = standard deviation of group 1 and group 2
 N_1 and N_2 = number of specimens in group 1 and group 2
 significant difference = yes if $t_{\text{actual}} > t_{0.995}$
 = no if $t_{\text{actual}} < t_{0.995}$

*See page 3 of this report.

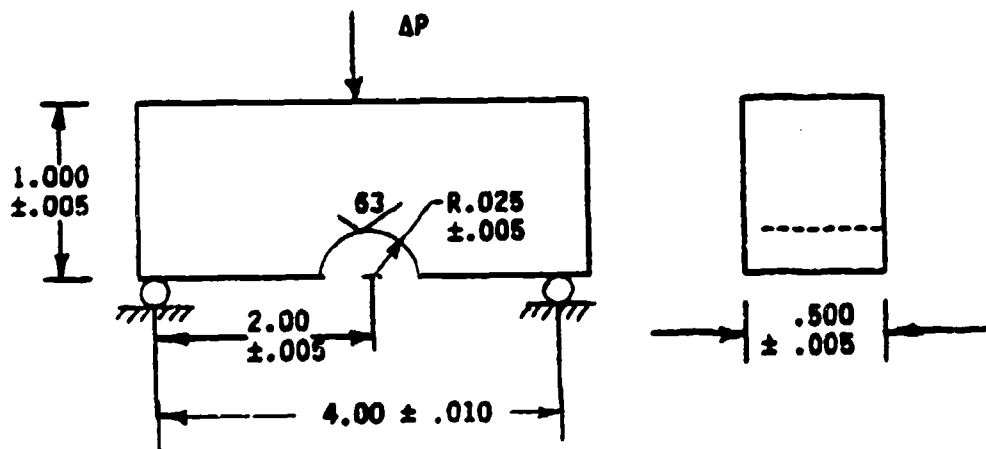
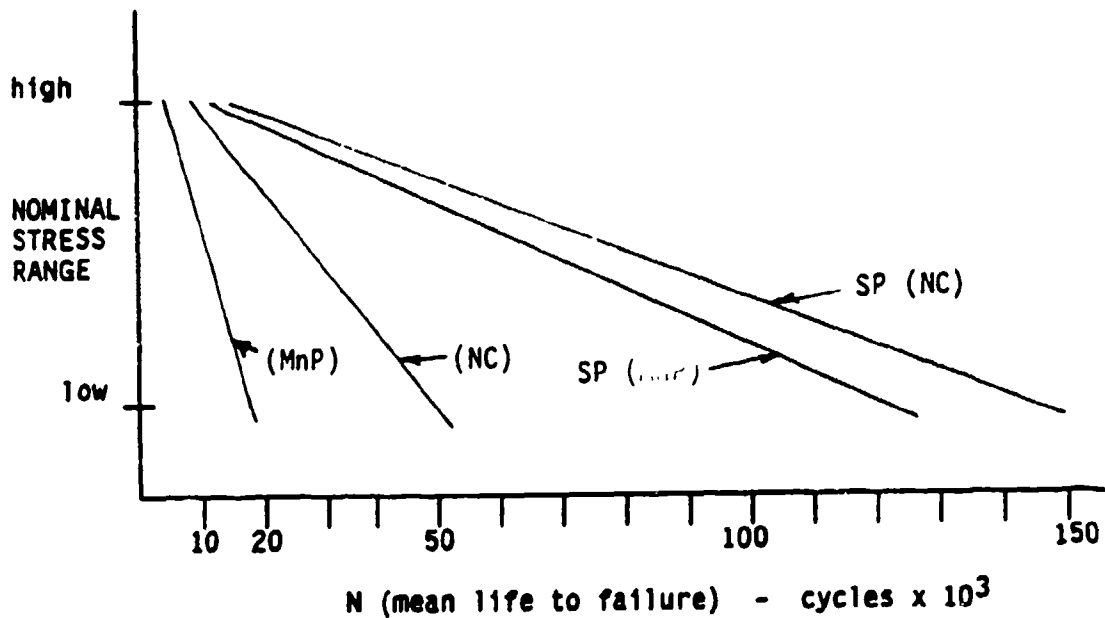


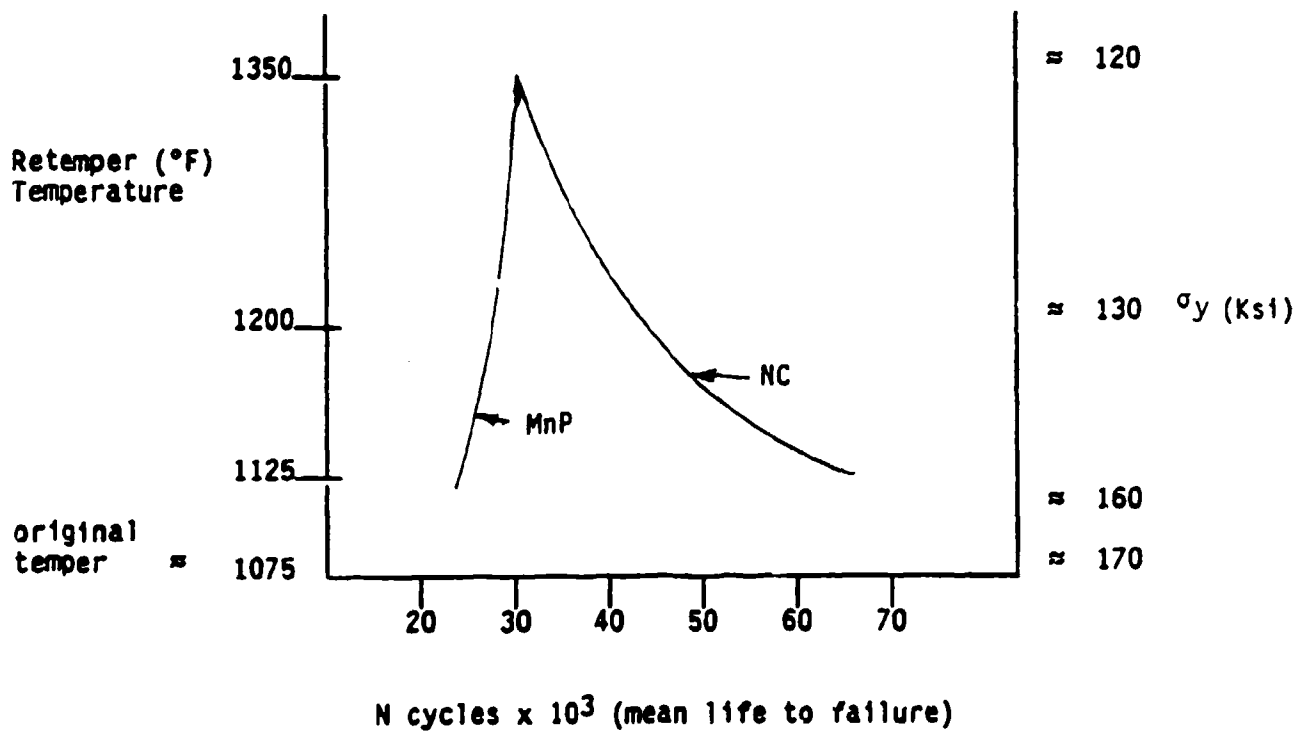
Figure 1. Three-point bend specimen design.



Low Stress - 11.7 to 117 Ksi
 High Stress - 17.0 to 170 Ksi

- SP (NC) = Shot peened - not coated
- SP (MnP) = Shot peened - manganese phosphate-coated
- (NC) = Not coated - not shot peened
- (MnP) = Manganese phosphate-coated - not shot peened

Figure 2. Nominal stress range versus crack initiation life. Shot peened (not coated versus manganese phosphate-coated) and not shot peened (not coated versus manganese phosphate-coated).



MnP = Manganese phosphate-coated.
NC = Not coated

Figure 3. Effect of material yield strength on crack initiation - not coated versus manganese phosphate-coated.



Depth of pits 0.003 to 0.004 inch.

Figure 4. Stress concentrations created by manganese phosphate.
Photomicrograph at 100X.

APPENDIX A

MANGANESE PHOSPHATING AND SHOT PEENING PROCEDURE

Manganese Phosphate Procedure

1. Clean with hot (180°F) alkaline detergent.
2. Hot water rinse.
3. Immerse in deruster (200°F).
4. Hot water rinse.
5. Immerse in condition M (180°F) which creates activation sites for the phosphating crystals.
6. Immerse in phosphate solution (30 minutes at 205°F).
Total acid 40 to 45 pts; free acid 6.5 to 7.5 pts (normal bath);
iron content 2 to 4 pts.
7. Cold water rinse.
8. Immerse in chromic acid (pH 2.5 to 3.5, 180°F).

Free Acid Pts - Number of ml of 0.1 normal sodium hydroxide to obtain a gray to greenish-gray color in 10 ml of phosphate solution.

Total Acid Pts - Total number of ml of 0.1 normal sodium hydroxide to obtain a pink color in 10 ml of phosphate solution.

Shot Peening Procedure

Eight specimens were sent to Metal Improvement Company, Windsor, CT, for shot peening. They used glass shot, size 330, and the Almen intensity was 0.006C to 0.008C per MIL-S-13165.

APPENDIX B

LOAD RANGE CALCULATIONS

The number of cycles (N) to initiate a crack can be obtained from the work of Underwood (ref 3) as

$$N = 95,000 (\sigma_m / \sigma_y)^{-3} \quad (1)$$

where σ_m is the maximum stress at the notch root and σ_y is the yield strength.

This equation fits the small radius (0.05 to 0.13 inch) data of Reference 3.

The maximum stress is given by Underwood as

$$\sigma_m = \frac{1.12 K_m}{\sqrt{\rho}} \quad (2)$$

where ρ is the notch radius (0.25) and K_m is the maximum stress intensity.

The equation for K_m is given in ASTM E-399 as

$$K_m = \frac{P_m \cdot L}{b w^{3/2}} f(a/w)$$

where L is the span (4.0), b is the specimen thickness (0.5), w is the specimen height (1.0), and f(a/w) is the crack depth factor.

$$f(a/w) = 3(a/w)^{1/2} \frac{\{1.99 - a/w(1-a/w)[2.15 - 3.93 a/w + 2.7(a/w)^2]\}}{2(1+2 a/w) \cdot (1-a/w)^{3/2}}$$

for

$$a/w = 0.25; f(a/w) = 1.34$$

hence

$$K_m = 10.72 P_m \quad (3)$$

Calculations for "High" and "Low" Load Ranges

High Load: Let $N = 20,000$ cycles

then, from Equation (1), $\sigma_m/\sigma_y = 1.68$

Let $\sigma_y = 170$ Ksi

then, from Equation (2), $K_m = 127.5 \text{ Ksi } \sqrt{\text{in.}}$

and $P_m = 11,890$ lbs

Used in tests: ΔP of 800 to 8000 lbs

Low Load: Let $N = 100,000$ cycles

then $P_m = 6960$ lbs

Used in tests: ΔP of 550 to 5500 lbs

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